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TECHNICAL REPORT

Radiological and Nuclear Detection Material Science: Novel Rare-Earth Semiconductors for Solid-State Neutron Detectors and Thin High-k Dielectrics

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Peter Dowben et al.

Prepared by:
University of Nebraska-Lincoln
312 N 14th Street
Lincoln, NE 68588

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Dowben, Peter; Belashchenko, Kirill; Losovyj, Yaroslav; Brand, Jennifer;

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University of Nebraska-Lincoln
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14. ABSTRACT

The goals have been to investigate new materials based primarily on rare-earth semiconductors, and explore new solid state detector configurations of these materials as neutron detectors. Seven materials (monoclinic and cubic Gd/HfO2 alloys, monoclinic and cubic Gd2O3, Gd/EuO, Gd doped GaN, and Li2B4O7) have been modeled, synthesized and made into devices. Various exemplar materials have been fabricated and the theoretical modeling has been completed for gadolinium oxide/hafnium alloys oxide the gadolinium oxides, gadolinium doped europium oxides and gadolinium doped GaN. Neutron detection testing has made for the Gd/HfO2 alloys to silicon devices, Gd2O3 heterostructures and, to a limited extent, lithium tetraborate. Characterization of heterojunction devices made from Gd/EuO alloys has been performed. The more recent efforts within the scope of the project have emphasized Li2B4O7 because this material's exceptional merits as a neutron detector material by itself and also because of the potential of this material for oxide-to-oxide heterostructures. GdNi alloys have also been studied.

15. SUBJECT TERMS

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UNIT CONVERSION TABLE

U.S. customary units to and from international units of measurement*

U.S. Customary Units	Multiply by → / ← Divide by†		International Units
Length/Area/Volume			
inch (in)	2.54	$\times 10^{-2}$	meter (m)
foot (ft)	3.048	$\times 10^{-1}$	meter (m)
yard (yd)	9.144	$\times 10^{-1}$	meter (m)
mile (mi, international)	1.609 344	$\times 10^{3}$	meter (m)
mile (nmi, nautical, U.S.)	1.852	$\times 10^{3}$	meter (m)
barn (b)	1	$\times 10^{-28}$	square meter (m^2)
gallon (gal, U.S. liquid)	3.785 412	$\times 10^{-3}$	cubic meter (m^3)
cubic foot (ft^3)	2.831 685	$\times 10^{-2}$	cubic meter (m^3)
Mass/Density			
pound (lb)	4.535 924	$\times 10^{-1}$	kilogram (kg)
unified atomic mass unit (amu)	1.660 539	$\times 10^{-27}$	kilogram (kg)
pound-mass per cubic foot (lb ft^{-3})	1.601 846	$\times 10^{1}$	kilogram per cubic meter (kg m^{-3})
pound-force (lbf avoirdupois)	4.448 222		newton (N)
Energy/Work/Power			
electron volt (eV)	1.602 177	$\times 10^{-19}$	joule (J)
erg	1	$\times 10^{-7}$	joule (J)
kiloton (kt) (TNT equivalent)	4.184	$\times 10^{12}$	joule (J)
British thermal unit (Btu) (thermochemical)	1.054 350	$\times 10^{3}$	joule (J)
foot-pound-force (ft lbf)	1.355 818		joule (J)
calorie (cal) (thermochemical)	4.184		joule (J)
Pressure			
atmosphere (atm)	1.013 250	$\times 10^{5}$	pascal (Pa)
pound force per square inch (psi)	6.984 757	$\times 10^{3}$	pascal (Pa)
Temperature			
degree Fahrenheit (^oF)	$[T(^oF) - 32]/1.8$		degree Celsius (^oC)
degree Fahrenheit (^oF)	$[T(^oF) + 459.67]/1.8$		kelvin (K)
Radiation			
curie (Ci) [activity of radionuclides]	3.7	$\times 10^{10}$	per second (s^{-1}) [becquerel (Bq)]
roentgen (R) [air exposure]	2.579 760	$\times 10^{-4}$	coulomb per kilogram (C kg^{-1})
rad [absorbed dose]	1	$\times 10^{-2}$	joule per kilogram (J kg^{-1}) [gray (Gy)]
rem [equivalent and effective dose]	1	$\times 10^{-2}$	joule per kilogram (J kg^{-1}) [sievert (Sv)]

*Specific details regarding the implementation of SI units may be viewed at http://www.bipm.org/en/si/.

†Multiply the U.S. customary unit by the factor to get the international unit. Divide the international unit by the factor to get the U.S. customary unit.

Accomplishments and New Findings

Background:

Specifically, the goals have been to investigate new materials based primarily on rare-earth semiconductors, and explore new solid state detector configurations of these materials as neutron detectors. Seven materials (monoclinic and cubic Gd/HfO_2 alloys, monoclinic and cubic Gd_2O_3, Gd/EuO, Gd doped GaN, and $Li_2B_4O_7$) have been modeled, synthesized and made into devices. Various exemplar materials have been fabricated and the theoretical modeling has been completed for gadolinium oxide/hafnium alloys oxide [6-8,10-15,31,36] the gadolinium oxides [11-13,34,35,37], gadolinium doped europium oxides [16-19,30,36] and gadolinium doped GaN [28,29,31,36,38]. Neutron testing has also been achieved for the Gd/HfO_2 alloy to silicon devices [10,14,15,31], Gd_2O_3 heterostructure diodes [37] and, to a limited extent, lithium tetraborate [42,43]. Characterization of heterojunction devices made from the Gd/EuO alloy has now also been reported by these investigators [30].

The more recent efforts (2011) within the scope of the project have emphasized $Li_2B_4O_7$ [20-27,32,33,39-44] because this material's exceptional merits as a neutron detector material by itself and also because of the potential of this material for oxide-to-oxide heterostructures [37]. One goal is the construction of an all-oxide diode, with appropriately doped materials, such as isotopically-enriched lithium tetraborate ($Li_2B_4O_7$). The all-oxide heterojunction device configuration should enhance device stability, signal-to-noise ratio, and may provide some single device kinetic energy resolution for low energy neutrons.

Gd/HfO_2 alloy semiconductor devices:

We have built several Gd/HfO_2 alloy on silicon heterojunction devices [6,8,10-11,13-15], as well as characterizing and modeling the Gd/HfO_2 alloy materials for future neutron device applications [31]. While Gd would be a p-type dopant, the oxygen vacancies in the alloy make the mixed oxide material n-type so a heterojunction diode can be formed with p-type silicon, with low concentrations level [6,8,10] and with n-type silicon at the higher Gd concentrations [6,10]. The films with Gd levels of (3, 10 and 15 at.%) in the Gd/HfO_2 alloy, were deposited on single crystal silicon (100) substrates using PLD [6-8,10-14] and a number of devices realized [6,8,10,14,15].

We have already been able to show that the 3% Gd/HfO_2 alloy forms the monoclinic structure and results in an excellent rectifying diode [6,8,10]. The 15% Gd/HfO_2 alloy forms the cubic or fluorite phase (and is also a better semiconductor in some respects according to our model band calculations) [6,11,12]. The 15% Gd/HfO_2 alloy based devices were seen to have an increase reverse bias current with an incident neutron flux but more importantly, single neutron pulse counting was obtained and is in good agreement with expectations from MCNP simulations [14,15]. To compare with theory, pulse height spectra were taken from 15% Gd HfO_2 on n-type silicon (100) heterojunction diodes, with electronic noise and signal suppression for pulses below 42 mV and with long time constants (5-10 microseconds), thus permitting many of the nuclear capture and decay resonances to be resolved in the pulse height spectra, by trying maximize charge collection per pulse [14,15]. Thermal neutron reactions with Gd nearly always result in a (n,γ) reaction, as in:

$^{157}Gd(n,\gamma) \rightarrow {}^{158}Gd + \gamma + \text{X-rays} + \text{IC } e^- + \text{ACK } e^-$

reactions which lead to the emission of low-energy gamma rays and internal (IC) and Auger (ACK) conversion electrons, as summarized in Figure 1. Gd does have a high internal conversion coefficient of nearly 39% for emitting a conversion electron but in fact less than 5% (3.4% + 1.4%) of all ^{157}Gd neutron capture events produce charged particles (internal conversion electrons) capable of generating electron-hole pairs in a Gd-based semiconductor device. Many of these conversion electrons occur through the decay channels creating 40 to 80 KeV pulses largely centered about the 72 KeV for ^{157}Gd(n,γ). Still much of the neutron capture cross-section is provided by the 16%-abundant ^{157}Gd isotope (255,000 barns) of natural abundance Gd. Enriched ^{157}Gd is commercially available and may be used to quintuple the absorption, if needed. The ^{157}Gd(n,γ)^{158}Gd and ^{155}Gd(n,γ)^{156}Gd reactions involve the emission of energetic gamma particles which might contribute to local energy transfer, as well as low-energy X-rays and conversion electrons. Still the relatively low energy of the conversion electrons produced by ^{157}Gd (30-40 times smaller compared to the daughter fragments of the ^{10}B nucleus) is the main drawback of using Gd as a neutron detector. The K-shell Gd Auger resonances, once the low energy pulses are electronically removed, can be resolved and compared with theory [14,15]. In spite of the large band gap of Gd/HfO_2 alloy, the signal to noise remains excellent, in part because of the very low leakage currents [14,15].

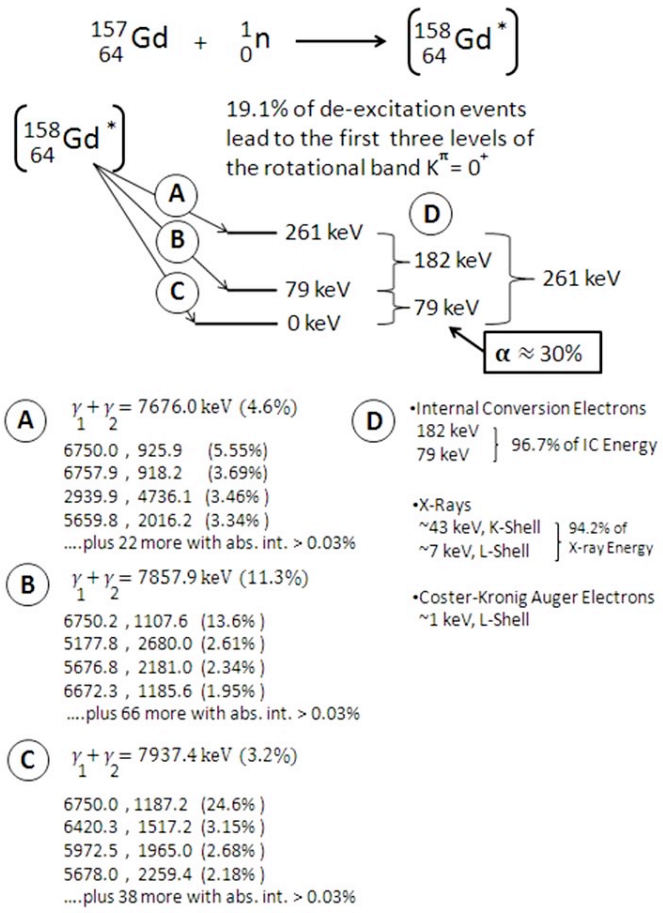

Figure 1: *The ^{157}Gd neutron capture branching ratios as summarized in [14].*

Gd_2O_3 semiconductor devices:

Monoclinic *n*-type [11-13,37] Gd_2O_3 on *p*-type silicon heterojunctions have been fabricated and the rectifying (diode like) properties of Gd_2O_3 to silicon heterojunctions have been demonstrated. The monoclinic Gd_2O_3 semiconductor devices were fabricated by pulsed laser deposition [11-13], while the cubic Gd_2O_3 has been fabricated on *p*-type silicon heterojunctions by supercritical fluid water deposition. Strong textured growth on Si(100) has been observed for cubic materials from this method [34,35]. Other techniques for fabrication were also explored: atomic layer expitaxy (ALD) [9].

For pulsed laser deposition of the monoclinic Gd_2O_3 on *p*-type silicon as well as the Mn doped $Li_2B_4O_7$ heterojunctions, the monoclinic Gd_2O_3 grows with a -402 texture growth [37].

This tends to suggest that the strong texture growth is somewhat insensitive to substrate. The various resulting Gd_2O_3 heterostructure device were tested by our colleagues at AFIT in a subcritical pile, and these devices seem to be sensitive to X-ray photoemission events. The Gd_2O_3 layers of these devices are too thin to have appreciable γ absorption, but Compton scattering, and bremsstrahlung scattering could easily produce enough X-rays to make the Gd_2O_3 layers sensitive to photoemission events, as is event by the increased number of counts evident with Cd shielding (which should decrease the number of counts if due to neutron events) and even more so with lead shielding [37], as seen in Figure 2. This makes the Gd_2O_3 based devices less desirable than solid state devices based on Gd doped GaN or devices based on Gd_2O_3-HfO_2 alloys (as described above). Steve McHale, in his doctoral thesis (AFIT, 2011) analyzed the ^{157}Gd neutron capture branching and decay processes, as outline in our work [14], and drew the compelling conclusion the effective figure of merit is better for Gd doped GaN, than Gd_2O_3 based solid state devices. Furthermore, we can now see that the Gd based semiconductor devices do not have a better figure of merit for neutron capture and neutron detection overall than boron based semiconductor devices, in spite of the higher neutron capture cross-sections. This result may be somewhat surprising, but remains a highly robust result nonetheless. The salient points of the analysis have been summarized by Lt. Col. Steve McHale, in his doctoral thesis (as also in Figure 1 of reference [14]) as follows:

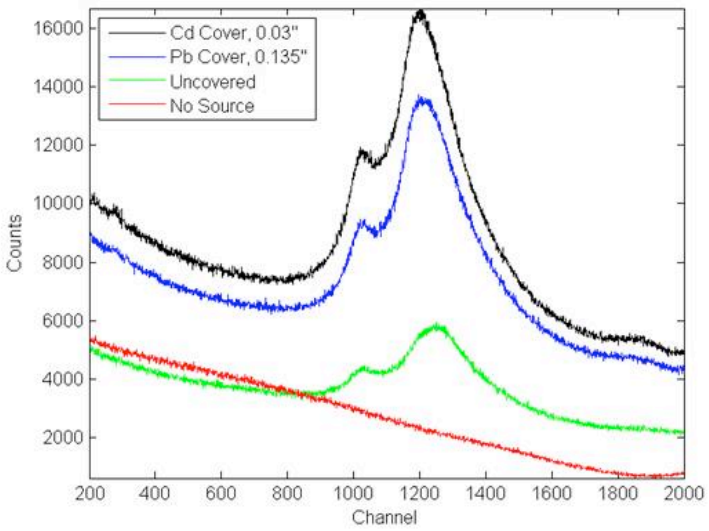

Figure 2: *The pulse height spectra of monoclinic Gd_2O_3 on Si(100) device showing an increase of signal events from a subcritical pile when the device is shielded by Cd (black). If these are neutron related events the signal should decrease, not increase. The increase is also greater with Pb shielding (blue), but not as much, because gamma attenuation by the lead foils will occur. This suggests that Compton scattered gamma to X-ray down conversion may be a problem in such devices and a possible significant contribution to the pulse height spectra [37]. These pulse height spectra (above) differ from the shape seen for neutron capture by gadolinium [14].*

(1) less than 5% (3.4% + 1.4%) of all ^{157}Gd neutron capture events produce charged particles (internal conversion electrons) capable of generating electron-hole pairs in a Gd-based semiconductor device,

(2) relatively low energy (keV-range) photon production occurs in

(a) the decay chain via emission of 43 keV **K**-shell and 7 keV **L**-shell characteristic X-rays, following internal conversion electron emission,

(b) the decay chain where 182 keV and 79 keV gamma rays are emitted for 3.2% (70% of 4.6%) and 7.9% (70% of 11.3%), respectively, of transitions,

(3) the dominant high energy photon (MeV-range) production occurs in decay chain, where 13.6% of 11.3%, or 1.5% of transitions produce coincident gamma rays of 6.7 MeV and 1.1 MeV.

A large band gap for Gd_2O_3 mean very few electron hole pairs are created by neutron capture and this will then compete with 43 keV **K**-shell and 7 keV **L**-shell and more outer Gd shell characteristic X-ray photoemission.

Gd/EuO alloy semiconductor devices:

We have now successfully fabricated 4% Gd/EuO alloy [16,30,38] and 4% Ce/EuO alloy [17] on *p*-type silicon heterojunctions. The structures were tested for rectifying (diode like) properties and in fact an Esaki tunnel diode [30] was fabricated. The structures were again fabricated by pulsed laser deposition. What is promising about this effort so far is the strong texture growths along <111> and the agreement between the theoretical and experimental band-structures [30]. This material was originally thought to have potential for oxide-to-oxide junctions, together with LiB_4O_7, but it now appears the Gd/HfO_2 alloy on silicon remains more advantageous because of the tendency that with Gd doping of EuO, the Gd/EuO alloy becomes metallic [30,38] and is generally not a phase stable material.

Gd doped GaN semiconductor materials:

In an effort to compensate for what we perceived as something of a failure in our efforts to develop Gd doped EuO, we also explored Gd doped GaN [27-29,38,40]. This line of materials development has proved to be very successful and shows considerable promise. The materials exhibited larger Schottky barrier heights [28] than seen for GaN and good hybridization of the rare earth atoms with the lattice [29] with the rare earths sitting substitutionally in the Ga site [27] was found. Recent work shows good agreement from density functional theory, for the placement of the occupied rare earth doped 4f states [38], as compared to the state placement determined from resonant photoemission [29]. This comparison of theory and experiment is now being written up for publication [40]. We believe these materials will provide better signal to noise with the smaller band gap of GaN (3.2 eV) as the number of electron-hole pairs created scale as a rough inverse to the band gap so that a 3.2 eV band gap is more advantageous than a 5.4 eV band gap (Gd_2O_3 and Gd:HfO_2). Among the Gd doped semiconductor device materials considered, Gd doped GaN also has the least stringent requirements for the device charge to voltage amplifier (roughly 1.2 fF). While solid state neutron detection, based on gadolinium capture, requires detecting a rather small charge, 0.1 to 1.2 fC, this nonetheless remains possible as the Schottky barrier heights with rare earth doped GaN are 50% higher than GaN [28], thus a decrease in leakage currents in reverse bias is anticipated with less background noise likely for the device, operating as a detector.

GdNi:

Almost all of our devices are based around a semiconductor diode structure, where the diode itself contains the neutron absorbing material, but the other device concept is to use a conversion layer material, where the neutron absorber in placed on one side of a more conventional p-n or p-i-n diode. A device based on coating a conventional diode with a neutron absorbing material is a conversion layer device. Obviously the problem is that if one uses gadolinium metal as the “conversion layer”, then over time, the gadolinium has the potential to oxidize, thus making gadolinium oxide Gd_2O_3, and ultimately adding to the total capacitance of

the device. This and other problems suggest that long term device stability, especially in harsh environments, is at risk. Alloys of gadolinium have the advantage that oxidation might be suppressed so that the alloy can still be used as an electrode as well as a neutron capture layer, with the capture fragments then detected in the more conventional diode, part of the overall detector device. Gd/Ni compounds with different Gd concentration were investigated by constant initial-state spectroscopy (CIS) [5] and we observed strong resonance photoemission of Gd 5*d* and 4*f* states that are quickly and strongly suppressed by increases in the nickel content of Gd/Ni alloy thin films. The screening of Gd ions by Ni 3*d* conduction electrons results in a dramatic suppression of the $5p^6 4f^7 5d^1 6s^2$ to $5p^6 4f^7 5d^2 6s^2$ resonant enhancement of photoemission valence band intensities indicating these alloys adopt short range order in the vicinity of each Gd atom and that the Gd 5*d* - Ni 3*d* and 6*s* - 4*s* states of Gd-Ni nearest neighbor atoms are strongly hybridized. This dressing of the gadolinium atoms within the alloy lattice by nickel will suppress gadolinium oxidation and make for a much more stable metallic gadolinium (alloy) layer.

Lithium tetraborate $Li_2B_4O_7$:

The most recent area of exploration has been $Li_2B_4O_7$ [20-27,32-33,39-44] with the ultimate target goal of preparation of an all-oxide device. The all-oxide configuration should enhance device stability, signal-to-noise ratio, and may provide some single device kinetic energy resolution for low energy neutrons. To that end, experimental and theoretical investigations of $Li_2B_4O_7$ have been carried out as part of a rather serious effort [20-27,32-33,39-44]. $Li_2B_4O_7$ is a compound for neutron detection applications because of both the lithium and boron and sensitivity increased readily because both ^{6}Li and ^{10}B enrich is easily achieved in these materials, and the bandgap can be tailored by the inclusion of metals such as Cu, Mn and Ag. Good quality V, Cu, Ag, Mn, Ce, Sm, Dy, Tb, Nd, Eu, Er containing crystals have now been fabricated. $Li_2B_4O_7$ crystals with ^{10}B or ^{6}Li enrichment have also been fabricated. The electronic structure of $Li_2B_4O_7$ was both calculated and measured by photoemission and surprisingly good agreement between theory and measurements has been demonstrated [24,25,33]. A key demonstration of the validity of theory efforts so far has been

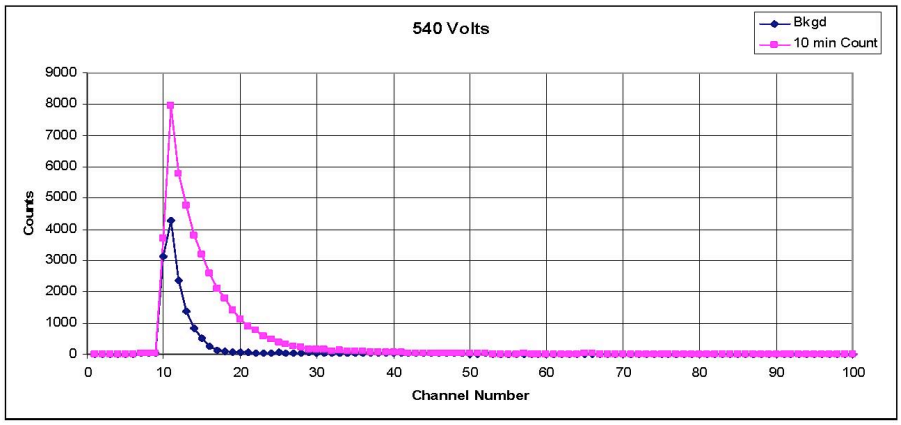

Figure 3: *The differential pulse height spectrum obtained with ^{10}B enriched $Li_2B_4O_7$ (100) with both neutron irradiation and for background, identified as typical noise spectrum. The crystal was placed in the radial neutron beam of the TRIGA Mark II nuclear reactor. The reactor power was set to 100kW (approximately 10^6 n/cm^2-s). The shutter to the beam was opened and the operating bias was increased positively until pulses were observed; the same was done for the negative bias. Once operating biases were obtained (540 V), 10 minute radiation measurements and 10 minute background measurements were obtained using a MCA. Courtesy of Kyle A. Nelson, Benjamin W. Montag, Douglas S. McGregor, Kansas State University.*

the success of theory [33] to reproduce the very different surface states that have been observed [21,24] at the $Li_2B_4O_7(110)$ and $Li_2B_4O_7(100)$ surfaces, and a valid comparison of the experimental and calculated optical properties of undoped lithium tetraborate [44]. Material characterization has been performed by extended X-ray adsorption fine structure, electron and optical spectroscopies, and electronic characterization, and lithium tetraborate has been used as a substrate for an all oxide $Gd_2O_3/Li_2B_4O_7$ device. Diodes have been fabricated.

While scintillation from neutron capture will certainly be an effective neutron detection scheme with lithium tetraborate [42], so far we have not yet extracted a clear pulse signal that can be associated with neutron capture in $Li_2B_4O_7$ (Figure 3). The neutron capture generated scintillation is far easier to establish, as even alpha irradiation will cause scintillation (Figure 4), as established by our colleagues at AFIT. This is significant because an alpha is one of the daughter fragments to ^{10}B or ^{6}Li capture of a neutron:

$$^{10}B + n \rightarrow {}^{7}Li\ (0.84\ MeV) + {}^{4}He\ (1.47\ MeV) + \gamma\ (0.48\ MeV)\ (94\%)$$

$$^{10}B + n \rightarrow {}^{7}Li\ (0.84\ MeV) + {}^{4}He\ (1.78\ MeV)\ (6\%)$$

and

$$^{6}Li + n \rightarrow {}^{3}H + {}^{4}He + (4.8\ MeV)$$

While compelling evidence of a charge pulse extraction due to neutron capture has not been obtained, as of the writing of this report, we believe that this problem can and will be addressed. It has been known that that lithium tetraborate could be used for neutron detection since 1966, but engineering these materials for use in a solid state neutron detectors has proven difficult, largely in part because of the large bandgap (9.7 eV). We have investigated the electronic structure of these materials but have gone on to demonstrate that with doping these materials could be part of very effective solid state neutron detectors. If we accept the mean free lifetime of the secondary charge carriers is $\tau = 50\ \mu s$ and the effective electron mass is $m^*/m_e = 0.15$, then the mobility may be estimated to be $\mu = e\tau/m^* = 1.8 \times 10^{14}\ cm^{3/2}\cdot g^{-1/2}$. Because of the very light mass image state [24] $m^*/m_e = 0.06$, the surface mobility may be estimated to be even larger, at about $\mu = e\tau/m^* = 4.4\times10^{14}\ cm^{3/2}\cdot g^{-1/2}$ [25]. Surprising, the mean carrier lifetime increases with increasing doping levels and likely, given the preliminary evidence so far available, even better for Mn doped lithium tetraborate than Cu doped lithium tetraborate. There are limits to possible improvements through doping: too much dopant material will also increase scattering decreasing the drift velocity substantially, but overall, this means that with the appropriate doping level, quality devices can be made. The experimental

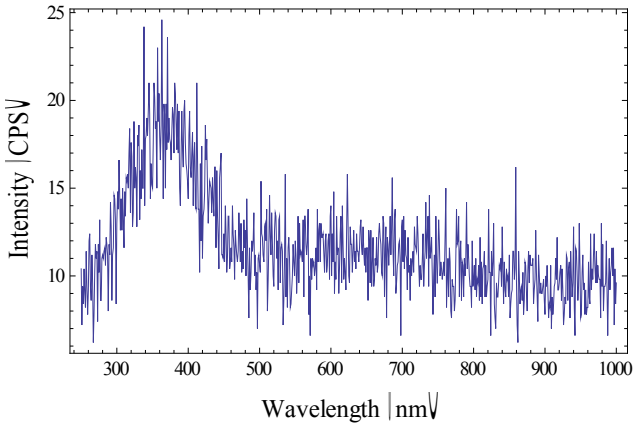

Figure 4. *Luminescence from undoped lithium tetraborate as a result of alpha irradiation from a ^{241}Am source. Unpublished.*

data [39] shows that manganese can be added successfully as a dopant and the XANES and EXAFS spectral features are characteristic of MnO bonds. Subsequent analysis of the experimental data [39], shows that manganese atoms likely substitute at boron sites, but occupation of the lithium sites are by no means excluded. In the former (B) site, the local bonds are severely strained and in the latter (Li) case, the data would support a picture that involves a large charge redistribution. The Mn doping of lithium tetraborate strongly resembles that of Mn doping of semiconducting boron carbide [4], but no evidence of pairwise doping of Mn is evident, as is the case in semiconducting boron carbide. The Mn K-shell near edge structure is strongly influenced by the fact that lithium tetraborate is a wide band gap insulator.

Because we have been dealing largely with single crystals of lithium tetraborate, radiation damage effects due to neutron irradiation and neutron capture have been characterized, and in the regard the neutron damage in both lithium tetraborate and lithium hexaborate can be compared [43]. This does result in the creation of a variety of displacement and vacancy defects (Figure 5), but in the low flux limit, there is little evidence of device degradation.

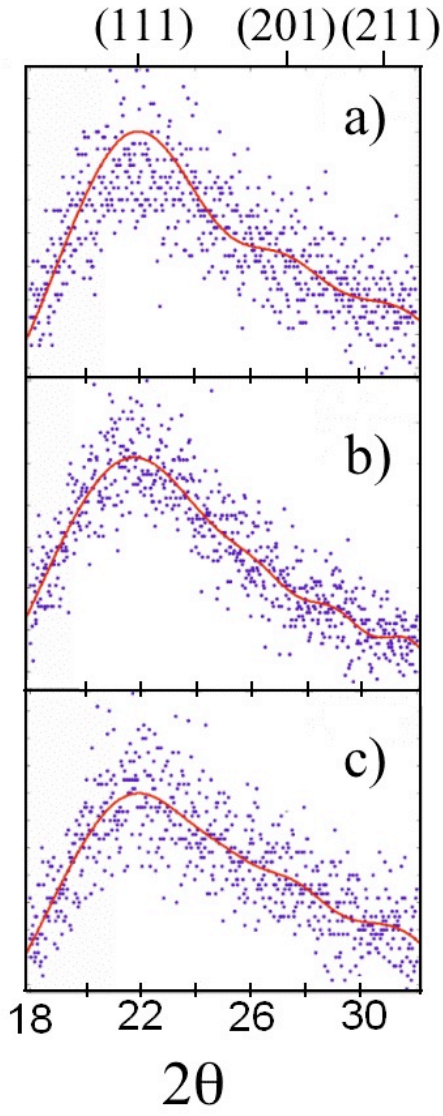

Figure 5: *Disorder introduced into microcrystalline lithium hexaborate (a) by radiation damage (b,c), with the neutron component of the radiation greatest in (c). The index of the diffraction peak is labeled at the top.*

Mn, Fe and Co-doped Boron Carbides:

The original proposal included a boron-rich and gadolinium-rich device for neutron detection. Semiconducting boron carbide was the obvious choice given the UNL expertise (Brand, Dowben). As part of our collaboration with the Air Force Institute of Technology, integral to this proposal, we began investigations of improvements to the semiconducting boron carbide material via metallic dopants [1-4]. Thus, this work should be considered "added value" for neutron detection, although not central to the original project.

Partly hydrogenated icosahedral boron carbide can be a very useful semiconductor material for neutron detection. The fabrication of homojunction solid state semiconductor devices from boron carbide has only recently been realized, although a semiconducting boron carbide has been sought since 1948. Prototype devices now include real-time solid-state neutron detectors, homojunctions and a variety of heterojunction diodes, including all boron-carbide heterojunction and heteroisomeric diodes. As part of the collaboration with Col. David LaGraffe at the Air Force Institute of Technology (now at NNSA), and his graduate students, we have explored Co [1-2], Fe [3] and

Mn [4] doped boron carbides. This work has resulted in the demonstration of cobalt [1,4] and Fe [3,4] doped boron carbide homojunctions. However, a claim of a device consisting of a Gd-rich layer on boron carbide would, at present, be premature.

Significance and relationships to goals:

All of the gadolinium based materials studied were part original goals of the proposal, except Gd doped GaN, and fit with the DTRA mission of better radiation detector materials and geometries for neutron detection for fissile material interception. The added value of the lithium borate investigations and studies of the Gd doped GaN, as additional advanced materials, furthers our basic collective understanding in solid state neutron detector materials. The new materials directions have great promise to achieve this goal of fabricating efficient and cost effects devices for solid state neutron detection. Indeed, the new materials investigated, lithium borates and Gd doped GaN, show perhaps better promise than monoclinic and cubic Gd/HfO_2 alloys, monoclinic and cubic Gd_2O_3, Gd/EuO, while monoclinic and cubic Gd/HfO_2 alloys are seen to be effective and have better figures of merit for use as neutron detector materials than monoclinic and cubic Gd_2O_3, Gd/EuO.

GdNi alloys are significantly more robust than Gd as a conversion layer material and possibly suitable for creating conducting ultrathin film radiation hard coatings for protection against neutron irradiation.

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37. Juan Colon Santana, Chris Young, Thomas McQuary, John McClory, Jim Petrosky, X. Wang, P. Liu, Jinke Tang. V.T. Adamiv, Ya. V. Burak, **P.A. Dowben**, "Growth of Monoclinic Gd_2O_3 Heterostructures", in preparation for submission to Radiation Measurements

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Executive Summary

Among the Gd doped semiconductor device materials considered, Gd doped GaN has the least stringent requirements for the device charge to voltage amplifier (roughly 1.2 fF) for single pulse neutron capture and detection, and likely the best overall figure of merit as a neutron detector device component, if the semiconductors are to be based on rare earth containing semiconductors. Gd doped GaN, while likely better as a neutron detector semiconductor material than Gd doped HfO_2, because of the smaller band gap (3.2 eV versus 5.4 eV) and better gamma rejection (Ga is smaller Z than Hf), both of these semiconductor materials (Gd:GaN and Gd:HfO_2) are demonstrably better for solid state neutron detection than Gd_2O_3 based solid state devices. It is also clear that the Gd doped GaN homojunction and heterojunction devices as well and the Gd/HfO_2 alloy on silicon heterojunction structures remains more advantageous than heterojunction structures based on Gd doped EuO. This is because of the tendency that with Gd doping of EuO, the alloy becomes metallic and because EuO does not have long term phase stability. Photoemission, resulting from scattered X-rays, contribute significant signal to Gd_2O_3 based solid state devices so that making such devices gamma blind presents a considerable challenge for these latter devices, in spite of the fact that the films of thickness sufficient to be opaque to thermal neutrons still have negligible gamma absorption at the gamma energies typical of fissile materials. There is now not clear guarantee that Gd based semiconductor devices will have a better figure of merit for neutron capture and neutron detection overall than boron based semiconductor devices, in spite of the higher neutron capture cross-sections of gadolinium over boron.

Summary of the Balaz Doctoral Thesis (UNL):
This works compares the molecular films of three different isomers of closodicarbadodecaborane (orthocarborane (1,2-$C_2B_{10}H_{12}$), metacarborane (1,7-$C_2B_{10}H_{12}$), paracarborane (1,12-$C_2B_{10}H_{12}$)) and two related icosahedral cage molecules, 1-phospha- 2-carbadodecaborane (1,2-$PCB_{10}H_{11}$) and 1-phospha-7-carbadodecaborane (1,7-$PCB_{10}H_{11}$) adsorbed on a variety of substrates. While the experimental electronic structure from combined photoemission and inverse photoemission studies of the molecular films are in good agreement with semi-empirical calculations for the isolated molecule, there is a shift in the chemical potential for each molecule. The experimental position of the molecular chemical potential implicates an influence of both interface and adsorbate dipole. The decomposition or dehydrogenation of the molecules was investigated as well. Decomposition results in a closing of the highest occupied molecular orbital (HOMO) to lowest unoccupied molecular orbital (LUMO) gap. This reflects a transition from the molecular film to a semiconductor. These molecules are precursors to different types of semiconductors (n or p-type). We find that some characteristics of the molecular films give us an insight into why they produce different types of semiconductors. Although the molecules are simply isomers of one another, a number of complexities are implicated in how the films are formed. Multiple polytypes of semiconducting boron carbides are indicated by this work, although the full range of polytypes has not been addressed.

access: http://0-proquest.umi.com.library.unl.edu/pqdweb?did=1280157391&Fmt=7&clientId=14215&RQT=309&VName=PQD

Summary of the Wooten Doctoral Thesis (AFIT):

Due to many of its attributes, $Li_2B_4O_7$ provides a possible material for incorporation as either a primary or companion material in future solid state neutron detectors. There is however a lack of fundamental characterization information regarding this useful material, particularly its electronic configuration. To address this, an investigation of $Li_2B_4O_7$(110) and $Li_2B_4O_7$(100) was undertaken, utilizing photoemission and inverse photoemission spectroscopic techniques. The measured band gap depended on crystallographic direction with the band gaps ranging from 8.9±0.5 eV to 10.1±0.5 eV. The measurement yielded a density of states that qualitatively agreed with the theoretical results from model bulk band structure calculations for $Li_2B_4O_7$; albeit with a larger band gap than predicted, but consistent with the known deficiencies of Local Density Approximation and Density Functional Theory calculations. The occupied states of both surfaces were extremely flat; to the degree that resolving periodic dispersion of the occupied states was inconclusive, within the resolution of the system. However, both surfaces demonstrated clear periodic dispersion within the empty states very close to theoretical Brillouin zone values. These attributes also translated to a lighter charge carrier effective mass in the unoccupied states. Of the two surfaces, $Li_2B_4O_7$(110) yielded the more consistent values in orthogonal directions for energy states. The presence of a bulk band gap surface state and image potential state in $Li_2B_4O_7$(110) was indicative of a defect-free surface. The absence of both in the more polar, more dielectric $Li_2B_4O_7$(100) was attributed to the presence of defects determined to be O vacancies. The results from $Li_2B_4O_7$(110) were indicative of a more stable surface than $Li_2B_4O_7$(100). In addition, Li 1s bulk and surface core level components were determined at the binding energies of -56.5±0.4 and -53.7±0.5 eV. Resonance features were observed along the [001] direction and were attributed to a Coster-Kronig process. Finally, the pyroelectric and piezoelectric character of $Li_2B_4O_7$ was explored more deeply and a non-zero, off-axis pyroelectric coefficient for the $Li_2B_4O_7$(110) direction was discovered.

access: http://www.dtic.mil/cgi-bin/GetTRDoc?AD=ADA521803

Summary of the McHale Doctoral Thesis (AFIT):

The thermal neutron capture cross section of the rare earth metal isotope ^{157}Gd is 255,000 barns and is the largest of all known natural elements, which distinguishes the material as a logical candidate for neutron detection. Semiconductors that incorporate large neutron absorption cross section materials, such as gadolinium, into their growth process are gaining attention, despite the relative immaturity of their development, and gallium nitride is one semiconductor material that has attained considerable recognition over the past two decades. However, there still exists an incomplete understanding of the physical and electronic properties of GaN materials, particularly whether even low concentrations of a rare earth in a GaN host can alter the surface electronic structure. To address this concern, investigations of the surface electronic structure and interface properties of GaN thin films doped with three distinct rare earths (Yb, Er, Gd) were undertaken using photoemission spectroscopy. The effective Debye temperatures of ytterbium and gallium in GaN:Yb thin films were obtained using X-ray photoemission spectroscopy. The vibrational motion normal to the surface resulted in a dimunition of photoemission intensities from which the effective Debye temperatures of 221 ± 30 K and 308 ± 30 K for Yb and Ga, respectively, were estimated. The similarity between the Yb and Ga Debye temperatures is indicative of a substitutional occupation of a Ga site by a Yb ion. The slightly

smaller effective surface Debye temperature for Yb correlates to a soft, strained surface, possibly due to an increased Yb–N bond length as compared to the Ga–N bond length.

The bonding with the GaN and the rare earth 4f hybridization were also examined. The 4d → 4f Fano resonances for various rare earth doped GaN thin films The thermal neutron capture cross section of the rare earth metal isotope ^{157}Gd is 255,000 barns and is the largest of all known natural elements, which distinguishes the material as a logical candidate for neutron detection. Semiconductors that incorporate large neutron absorption cross section materials, such as gadolinium, into their growth process are gaining attention, despite the relative immaturity of their development, and gallium nitride is one semiconductor material that has attained considerable recognition over the past two decades. However, there still exists an incomplete understanding of the physical and electronic properties of GaN materials, particularly whether even low concentrations of a rare earth in a GaN host can alter the surface electronic structure. To address this concern, investigations of the surface electronic structure and interface properties of GaN thin films doped with three distinct rare earths (Yb, Er, Gd) were undertaken using photoemission spectroscopy. The effective Debye temperatures of ytterbium and gallium in GaN:Yb thin films were obtained using X-ray photoemission spectroscopy. The vibrational motion normal to the surface resulted in a dimunition of photoemission intensities from which the effective Debye temperatures of 221 $\pm$ 30 K and 308 $\pm$ 30 K for Yb and Ga, respectively, were estimated. The similarity between the Yb and Ga Debye temperatures is indicative of a substitutional occupation of a Ga site by a Yb ion. The slightly smaller effective surface Debye temperature for Yb correlates to a soft, strained surface, possibly due to an increased Yb–N bond length as compared to the Ga–N bond length. The bonding with the GaN and the rare earth 4f hybridization were also examined. The 4d → 4f Fano resonances for various rare earth doped GaN thin films

access: www.dtic.mil/dtic/tr/fulltext/u2/a549030.pdf

Summary of the Juan Colon Doctoral Thesis (UNL):

This works compares the electronic structure of various rare earth oxides: Gd_2O_3, Gd doped EuO, Ce doped EuO, and Gd doped HfO_2. The rare earth doped EuO materials are seen to be metallic while the films gadolium oxide and hafnium oxide are dielectrics. Devices with rectifying behavior have been made from heterojunctions with silicon for all of the named oxides, with the exception of Gd doped EuO where tunnel diode like behavior was observed. High quality films of EuO and $Eu_{0.96}Gd_{0.04}O$ were grown on p-type Si(100) via pulsed laser deposition. X-ray diffraction results show that the addition of Gd changes the growth texture from [001] to [111]. Angular-resolved photoemission spectroscopy reveals electron pockets around the X points in Gd-doped EuO, indicating that the band gap in EuO is indirect. Combined photoemission and inverse photoemission measurements show an apparent transition from an *n*-type to *p*-type behavior, which is likely due to band bending near the polar (111) surface. The influence of magnetic field on the diode rectifying behavior was observed for Gd doped EuO and chromium carbide alloyed with carbon in a diamond like film heterojunction with silicon.

Training and Professional Development

Research conducted in the laboratories of Prof. Jennifer Brand and Prof. Peter Dowben has resulted in the graduation of two Ph.D. graduate students from the University of Nebraska – Lincoln, while contributing significantly to the training of other graduate students, including two

Ph.D. students from the Air Force Institute of Technology, and several M.Sc. student again from Air Force Institute of Technology as well as several undergraduates. The following students completed advanced degrees submitting theses directly related to the objectives of improving neutron detection with advanced materials and devices, directly related to the goals and outcomes of this project. Another student is expected to graduate in early 2012 (Juan Colon – winner of 2011 Leo Falicov Prize from the American Vacuum Society).

Students Graduated:

Snjezana (Snow) Balaz, Ph.D., College of Engineering, University of Nebraska, 2007; M. Sc. Department of Physics and Astronomy, University of Nebraska
Now post-doctoral associate, Department of Physics, University of California - Riverside
Thesis: "Electronic Structure and Evolution of Dehydrogenation of orthocarborane, metacarborane, paracarborane, orthophosphacarborane, and meta-phosphacarborane"
Student prizes: AVS Dorothy M. and Earl S. Hoffman Travel Grant (2003); Nebraska chapter of Sigma Xi travel grant (2004)

Major Lonnie Carlson (U.S. Army), M.Sc., Department of Engineering Physics, Graduate School of Engineering and Management, Air Force Institute of Technology, Air University Air Education and Training Command, March 2007
Now: Weapon Concepts team chief for the Global Strike requirements division at STRATCOM
Thesis: "Cobalt Doping Of Semiconducting Boron Carbide Using Cobaltocene"

Lt. Bryan Blasey (US Air Force), M.Sc., Department of Engineering Physics, Graduate School of Engineering and Management, Air Force Institute of Technology, Air University Air Education and Training Command, March 2008
Now: Weapon Concepts team chief for the Global Strike requirements division at STRATCOM

Markus Natta, Ph.D., College of Engineering, University of Nebraska, 2008; Post-doctoral associate, College of Engineering, University of Nebraska May 2009. Now, faculty on St. Kitts.
Thesis: "Fabrication and Characterization of Novel Boron and Gadolinium Rich Power Generation and Real-Time Neutron Detection Materials and Devices"

Lt. Col. David Wooten, (U.S. Army) Ph.D. 2010, Graduate School of Engineering and Management, Air Force Institute of Technology,
Now: staff STRATCOM, Offutt AFB, Omaha, NE
Thesis: "Electronic Structure Of Lithium Tetraborate"

Lt. Col. Stephen R. McHale, (U.S. Army) Ph.D. 2011, Graduate School of Engineering and Management, Air Force Institute of Technology,
Now: now faculty, Air Force Institute of Technology
Thesis: "The Effects of Rare Earth Doping on Gallium Nitride Thin Films"

Students Soon to Graduate:

Juan Colon-Santana, Department of Electrical Engineering, University of Nebraska
Student prizes: Richard Larson Fellowship (2007-2008); Avery Fellowship (2008); Leo Falicov Prize from the American Vacuum Society (2011)

Dissemination of the Results to Communities of Interest

Results have been submitted primarily by publications in refereed technical journals, and by presentations. These are listed below. While much of this activity has been aimed at the Neutron Detection Community, there is increasing interest on the part of the semiconductor community and rare earths and actinides community, as indicated by an invited talk (Dowben) at several national and international conferences.

A total of 32 **peer-reviewed** archival publications are in print or in press, as listed below. In addition, 3 manuscript are under review and 10 more manuscripts are presently in preparation and near completion. We have also filed 3 provisional patents.

Papers published, or submitted:

Publications

1. L. Carlson, D. LaGraffe, Snjezana Balaz, A. Ignatov, **Ya. B. Losovyj**, J. Choi, **P.A. Dowben**, and **J.I. Brand**, "Doping of boron carbides with cobalt, using cobaltocene", *Applied Physics A* **89** (2007) 195-201

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Presentations (invited talks denoted by *):

1. "Optimization of 3m TGM beamline performance at CAMD", Synchrotron Radiation Instrumentation (SRI2007), April 26, 2007, Baton Rouge, Louisiana [presented by I. Ketsman]
*2. "Using a synchrotron to study and fabricate cluster assembled semiconductors", Swiss Light Source, Paul Scherrer Institue, July 6, 2007 Invited
*3. "The n-type and p-type Gd/HfO2 alloy to silicon heterojunction diode", 3rd International Workshop on Surface Physics: Nanostructures on Surfaces, Polanica-Zdroj, Poland, 13 September 2007 [presented by I. Ketsman]
*4. "Using a synchrotron to study and fabricate cluster assembled semiconductors", Electronics Dept., Lviv National University, 50 Dragomanov Str., 79005 Lviv, Ukraine, colloquium, 18 September 2007 Invited
5. "Local Environment and Anisotropy of Co in $B_{1-x}C_x$", 52nd Conference on Magnetism and Magnetic Materials, November 8, 2007, Tampa, Florida [presented by R.Skomski]
6. "The effect of Gd doping on the atomic and electronic structure of HfO2 thin films", 2008 APS March Meeting March 12, 2008; New Orleans, Louisiana, Abstract: P36.00004 [presented by Ihor Ketsman]
*7. "Photo-fragmentation of the closo-carboranes", Boron in the Americas XI, St. Louis University, June 8; St Louis, Mo. [presented by Jing Liu]
*8. "The History and Development of Boron Carbon Alloy Devices and Rare Earth Heterojunction Neutron Detectors", Applied Physics and Nuclear Engineering Seminar, Air Force Institute of Technology, Dayton, Ohio, July 11, 2008 Invited

*9. "Correlation effects in gadolinium doped cluster molecules and compounds: comparison of theory and experiment", XXXII International Conference of Theoretical Physics: Coherence and correlations in nanosystems, Ustron, Poland, Sept. 8, 2008 Invited
10. "Comparison of Gd2O3 and Gd/HfO2 alloy Electronic Structure", MRS Fall Meeting, Boston, December 3, 2008 [presented by Dave Wooten] abstract D7.16, David J Wooten, Juan Colon-Santana, N. Lozova, James Petrosky, A. Sokolov, Jinke Tang, Wendong Wang, I. Ketsman and Peter Dowben
*11. "The History and Development of Boron Carbon Alloy Devices and Rare Earth Heterojunction Neutron Detectors", Physics and Energy Engineering Seminar, University of Wyoming, Laramie, Wyoming, December 5, 2008 Invited
12. "Theoretical and Experimental Efficiency Comparison of 15% Gd-doped HfO2/Silicon Heterojunction", Abstract: L1.4, 2009 Spring Metting of the MRS, San Francisco CA, April 14, 2009 [presented by James Petrosky]
13. "The Band Structure of $Li_2B_4O_7$", Abstract: L4.4, 2009 Spring Meeting of the MRS, San Francisco CA, April 14, 2009 [presented by David Wooten] "Novel Rare Earth Semiconductors", 2010 LSU-CAMD User Meeting, April 16, 2010, CAMD [presented by Juan Colon]
*14. "Novel Rare Earth Semiconductors", 2010 LSU-CAMD User Meeting, April 16, 2010, CAMD [presented by Juan Colon]
*15. Novel Semiconducting Materials for Real-Time Neutron Detection" Jennifer I. Brand University of Wyoming, Laramie, Wyoming. 14 January 2010 **Invited**
16. "Semiconducting Gadolinium Oxide Materials as Real-Time Neutron Detectors" Jennifer I. Brand ACS National Meeting 25 March 2010
17. "Real-Time Neutron Detection" Jennifer I. Brand**,** Nov 9, 2009 to UNL visitors from Booz, Allen and STRATCOM
18. "Real-Time Neutron Detection" Jennifer I. Brand**,** Mar 12, 2010 to UNL visitors from STRATCOM
19. "User-centered Task-specific Real-time Neutron Detection Instrumentation". Jennifer I. Brand **Invited** July 28, 2010 to UNL visitors from The Pentagon
20. "Temperature-composition phase diagrams of Gd-doped EuO and EuS". J. M. An, S. Barabash, and K. D. Belashchenko, 2009 APS March Meeting, Pittsburgh, Pennsylvania, March 16-20, 2009.
*21. "Correlation effects in gadolinium compounds: comparison of theory and experiment", Seminar, Paul Scherrer Institute, Switzerland, Wednesday Sept. 29th. 2010
22. "Photoemission Study of Au-Schottky Barrier Formation on YbGaN Thin Films using Synchrotron Radiation", AVS 57th International Symposium & Exhibition, Albuquerque, NM, Oct. 18, 2010 paper AC+TF-MoA8 [presented by Steve McHale]
23. "Effects on the Electronic Band Structure of EuO Films upon Gd doping", AVS 57th International Symposium & Exhibition, Albuquerque, NM, Oct. 18, 2010 paper EM+MI-MoA8 [presented by Juan Colon]
*24. "Correlation Effects in Gadolinium Compounds: Comparison of Theory and Experiment", AVS 57th International Symposium & Exhibition, Albuquerque, NM, Oct. 19, 2010 paper AC+MI-TuM3 (Dowben: invited)
25. "Electrical, Optical, and Magnetic Properties of Gd-doped GaN", Spring Meeting of the MRS, Symposium V: Rare-Earth Doping of Advanced Materials for Photonic Applications, San Francisco, CA, April 28 - 29, 2011; abstract: V3.7, Adriana M. Rivera, Javier Wu, Hannu Huhtinen, Wojciech M. Jadwisienczak, Ratnakar Palai, Steve McHale and Peter A. Dowben [presented by Adriana M. Rivera]
26. "Electrical and Optical Properties of Er Doped GaN and AlN Thin Films and Superlattices", Spring Meeting of the MRS, Symposium V: Rare-Earth Doping of Advanced Materials for Photonic Applications, San Francisco, CA, April 28 - 29, 2011; abstract: V3.7, Kiran Dasari, R. Acevedo Esteves, Steve McHale, J. Wu, H. Huhtinen, Peter Dowben and R. Palai [presented by Kiran Dasari]

27. "Alterations in the Electronic Band Structure and Magnetic Properties of EuO Films via Rare Earth Doping", AVS 58th International Symposium, November 3, 2011, Nashville, TN, paper MI-WeA-12 [presented by Juan Colón Santana]
28. "The Surface States of Lithium Tetraborate", AVS 58th International Symposium, November 3, 2011, Nashville, TN, paper SS-ThA-11
29. "Supercritical Production of Cubic Gadolinium Oxides for Real-Time Neutron Detection", 10th International Symposium on Supercritical Fluids, May 16, 2012, San Francisco, CA, Peter Molnar and Jennifer I. Brand

New Discoveries, Inventions, or Patent Disclosures

3 provisional patents filed:

1. Anthony Caruso, James C. Petrovsky, John W. McClory, Peter A. Dowben, William Miller, Thomas Oakes, Abigail Bickley, "Apparatus And Method For Directional And Spectral Analysis Of Neutrons", US Provisional Patent Application Serial No. 61/274,753 filed 08/20/09 for UM Disclosure No. 10UMK002; complete patent application filed 8/20/2010

2.&3. Jinke Tang, Peter Dowben, David Wisbey, Ihor Ketsman, Jennifer Brand, Andre Sokolov, Yaroslav Losovyj, "Neutron Detection Using Gd-Loaded Oxide and Nitride Heterojunction Diodes" U.S. Patent Application Serial No: 61/505,229; U.S. Patent Application Serial No: 61/505,223, Filed July 7, 2011

Participants

Faculty/Principle Investigators:

Jennifer I. Brand, Professor, College of Engineering and Nebraska Center for Materials and Nanostructures, 245N WSEC, University of Nebraska, Lincoln, NE 68588-0511, e mail: jbrand@unl.edu; (402) 472-9320

Peter A. Dowben, Charles Bessey Professor of Physics, Nebraska Center for Materials and Nanoscience, Department of Physics and Astronomy, Theodore Jorgensen Hall, 855 North 16th Street, University of Nebraska-Lincoln, Lincoln, NE 68588-0299, tel: 402-472-9838, FAX: 402-472-6148 , e mail: pdowben@unl.edu

Kirill D. Belashchenko, Associate Professor, Nebraska Center for Materials and Nanoscience, Department of Physics and Astronomy, Theodore Jorgensen Hall, 855 North 16th Street, University of Nebraska-Lincoln, Lincoln, NE 68588-0299 , e mail: kdbel@unlserve.unl.edu

Yaroslav Losovyj, Adjunct Professor, The J. Bennett Johnston Sr. Center for Advanced Microstructures and Devices (CAMD), Louisiana State University, Baton Rouge, LA and the Nebraska Center for Materials and Nanostructures, e mail: ylosovyj@lsu.edu

Post-doctoral support:
Ildar Sabirianov (engineering: now faculty at S.U.N.Y. Oswego)
Snow Balaz (engineering: post-doc at Ohio State)
Ihor Ketsman (physics)
Siqi Shi (physics: Academica Sinica)

Joonhee Michael An (physics)
Marcus Natta (engineering: now Faculty, St. Kitts)
J. Xiao (physics: now University of Erlangen)
Peter Molnar (engineering)

Graduate Students:
Marcus Natta (engineering: now Faculty, St. Kitts)
Pamela Jeppson (engineering)
Rebecca Lacy (engineering)
Jing Liu (physics: now LSU)
David Wisbey (physics: now NIST)
Juan Colon (engineering)
Lt. Col. Lonnie Carlson (engineering)
Lt. Bryan Blasey (AFIT)
Lt. C. David Wooten (AFIT: now STRATCOM)
Maj. Christopher M. Young (AFIT)
Lt. Col. Steve McHale (AFIT: now AFIT faculty)
Maj. Christina Dugan (AFIT: now faculty at the Air Force Academy)

Other Organizations

Air Force Inst. Technology
National Nuclear Security Agency
West Virginia University
Univ. Wyoming
Sigma-Aldrich
Univ. of Lviv
Univ. of Puerto-Rico
Kansas State University
U. Missouri K.C.
Arizona State University

Important External Collaborators

Prof. James Petrosky (Air Force Inst. Tech.)
Lt. Col. John McClory (Air Force Inst. Tech./DTRA)
Col. David LaGraffe (Air Force Inst. Tech./NNSA)
Prof. Bob Hengehold (Air Force Inst. Tech.)
Prof. Larry Halliburton (West Virginia University)
Prof. Jinke Tang (Univ. Wyoming)
Prof. Neil M. Boag (Sigma-Aldrich)
Prof. Yaroslav Burak (Univ. of Lviv)
Prof. R. Palai (Univ. of Puerto-Rico)
Prof. Douglas McGregor (KSU)
Prof. Tony Caruso (U. Missouri K.C.)
Prof. Mark van Schilfgaarde (Arizona)

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FT. BELVOIR, VA 22060-6201
ATTN: DTIC/OCA

DEPARTMENT OF DEFENSE CONTRACTORS

QUANTERION SOLUTIONS, INC.
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KIRTLAND AFB, NM 87117-5669
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